Title: Hydrothermal vs. sol-gel auto combustion: A comparative study of structural and magnetic properties in graphene oxide-$CoNiFe_2O_4$ nanocomposites

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Abstract: GO-$CoNiFe_2O_4$ (GO-CNF) nanocomposites of different compositions such as GO: CNF (1:2), GO: CNF (1:1), GO: CNF (2:1) have been synthesized through in-situ sol-gel auto combustion and hydrothermal method. The synthesized GO, and GO-CNF nanocomposites were characterized by X-ray Diffraction (XRD), Fourier-Transform Infrared Spectroscopy (FTIR), Thermogravimetric analysis (TGA), Physical Properties Measurement System (PPMS), Transmission Electron Microscopy (TEM), and Raman Spectroscopy to study their comparative properties. The successful synthesis of GO and GO-CNF nanocomposites in both methods was revealed by XRD analysis. The presence of functional groups containing oxygen in GO and other bonds in GO-CNF nanocomposites was confirmed through FTIR, which showed good bonding between GO and CNF in the nanocomposites. The PPMS study of the samples synthesized by both sol-gel auto-combustion and hydrothermal methods shows the ferrimagnetic properties of spinel ferrites-based GO nanocomposites. The thermal stability of the nanocomposites was determined by TGA. The Raman spectrum provided insights into defects, carbon sp^2 vibrations, and the stacking order of the samples. The microscopic morphologies of GO and GO-$CoNiFe_2O_4$ nanocomposites were characterized by TEM. Multilayered GO sheets with ~0.98 nm thickness were revealed by Atomic force microscopy (AFM) analysis. Energy-Dispersive X-ray (EDX) spectroscopy and High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) confirmed the uniform distribution of CNF in the GO sheet. Overall, the sol-gel route yields stronger GO-CNF interfacial interaction and improved performance compared to the hydrothermal method, reflected by noticeably lower thermal weight loss (6% for SFG12 vs 37% for HFG12) and higher saturation magnetization (Ms = 35.14 emu/g for SFG12 vs 23.42 emu/g for HFG12). The Sol-gel method was found to be a better synthesis method compared to the hydrothermal method. Amongst all the applied compositions, GO: CNF = 2:1 portrayed the best performance in terms of thermal stability, crystallinity, as well as homogeneous interfacial distribution of the nanocomposite.

Key words: Graphene oxide, Spinel ferrite,Nanocomposite, In-situ sol-gel auto combustion, Hydrothermal

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